

# HOT PRESSING AND ITS DEFORMATION MECHANISM OF Ag-SHEATHED DPSCCO SUPERCONDUCTING TAPES<sup>①</sup>

Ma Yanwei, Wang Xianjin and Wang Zutang\*

*Department of Metal Forming,*

*University of Science and Technology Beijing, Beijing 100083, P. R. China*

*\* Tsinghua University, Beijing 100084, P. R. China*

**ABSTRACT** Experiments of hot pressing of the tapes were carried out. Parameters of pressure, temperature and time were determined. Deformation mechanism of hot pressing were also studied. Experimental results showed that hot pressing caused the core to become more dense and could avert the formation of cracks effectively. At 77 K and zero field, the maximum  $J_c$  value was up to  $2.25 \times 10^4$  A/cm<sup>2</sup> under the conditions of 750 °C, 200 MPa and 30 min.

**Key words** Ag-sheathed superconducting tapes hot pressing deformation mechanism critical current density

## 1 INTRODUCTION

The “pressing-sintering” method is frequently applied to fabricating the silver-clad Bi (2223) tapes. The higher  $J_c$  in pressed tape can be attributed to a higher core density and better grain alignment. On the other hand, cold pressing also destroys the connections between the grains. Increasing the deformation temperature may avoid this harmful effect. Several techniques such as hot pressing, hot rolling and hot forging have been demonstrated as effective ways of achieving dense 2223 phase ceramics with a high degree of grain alignment<sup>[1-5]</sup>. The highest  $J_c$  value of hot rolled tape was  $1.8 \times 10^4$  A/cm<sup>2</sup> (77 K, zero field)<sup>[4]</sup>. For bulk samples, at 77 K and self-field transport  $J_c$  of 11 500 A/cm<sup>2</sup> and 10 000 A/cm<sup>2</sup> were obtained by hot pressing<sup>[1]</sup> and hot forging<sup>[2]</sup>, respectively. It is worthwhile mentioning that we present the first results of silver-sheathed Bi(2223) tapes treated by hot pressing<sup>[5]</sup>, and a critical current density

of  $2.25 \times 10^4$  A/cm<sup>2</sup> (77 K, zero field) was measured on tapes. In this paper, hot pressing and its deformation mechanism of Ag-sheathed Bi (2223) superconducting tapes were studied in detail.

## 2 EXPERIMENTAL

The precursor powders with a composition of Bi<sub>1.84</sub>Pb<sub>0.34</sub>Sr<sub>1.91</sub>Ca<sub>2.03</sub>Cu<sub>3.06</sub>O<sub>x</sub> were prepared from Bi<sub>2</sub>O<sub>3</sub>, PbO, SrCO<sub>3</sub>, CaCO<sub>3</sub> and CuO. The powders were mixed and calcined at 800 °C for 72 h in air with several intermediate grindings<sup>[6]</sup>. Then the powders were packed into silver tubes, and the tubes were drawn into round wire with a diameter of 1.0 mm and rolled into tapes with thickness of 0.1 mm. These tapes were cut into short tapes which were treated by cold pressing (CP) and hot pressing (HP), respectively. Then the pressing and annealing process were repeated for several times. The experimental details are illustrated in Ref. [5].

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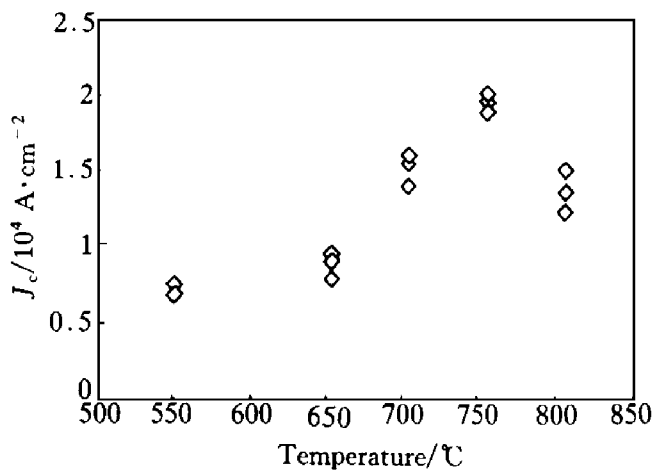
The  $J_c$  measurements were performed by conventional four-probe DC method at 77 K using a criterion of 1  $\mu\text{V}/\text{cm}$ . XRD was performed by peeling off the Ag sheaths from one side and exposing the flat ceramic core surface to the X-ray beam. The SEM analysis was performed on the fracture surface of both longitudinal and transverse cross-sections of the tape without the Ag-sheath.

### 3 DETERMINATION OF HP PARAMETERS

#### 3.1 HP temperature

Fig. 1 shows the effects of HP temperature on critical current density under pressure of 200 MPa for half an hour. The critical current increased slightly for the samples hot pressed at temperatures below 650 °C. For temperature above 650 °C, critical current density increased steadily. Further increasing temperature above 800 °C,  $J_c$  decreased gradually.

According to our experimental results, the best HP temperature appears to be located just around 750 °C in air.



**Fig. 1 HP temperature versus critical current density curves**

The tapes after the process of 838 °C, 50 h+ HP+ 838 °C, 60 h+ HP+ 838 °C, 60 h, here HP indicates the tapes were pressed under 200 MPa for half an hour at a temperature of 550 °C, 650 °C, 700 °C, 750 °C and 800 °C, respectively.

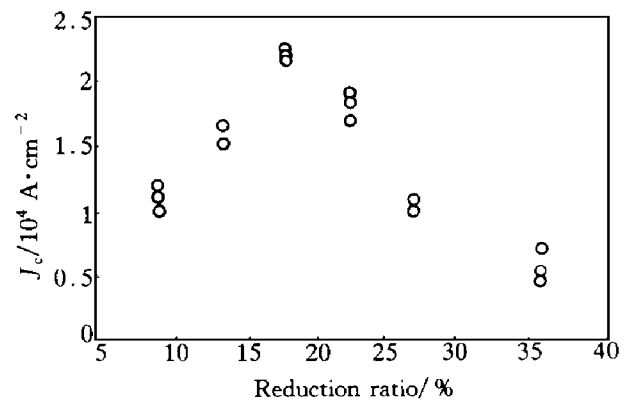
#### 3.2 HP pressure

For the optimization of HP pressure the temperature was kept at 750 °C for 30 min. The

HP reduction ratio of different pressure versus critical current density curves are shown in Fig. 2. From Fig. 2, we can see that  $J_c$  increases with the increase of reduction ratio. The  $J_c$  values reaches the highest when the pressure is 200 MPa, namely the reduction ratio is about 20%. When higher pressure was applied, we found that the surface quality of the tape became worse and  $J_c$  was usually lower.

#### 3.3 HP time

In order to determine HP time, the effects of time 10, 15, 30, 35 and 40 min, respectively, on  $J_c$  under the conditions of 750 °C and 200 MPa were studied. Results showed that  $J_c$  values were not high if HP time was shorter than 15 min or longer than 35 min<sup>[7]</sup>. The reasonable HP time was about 30 min.



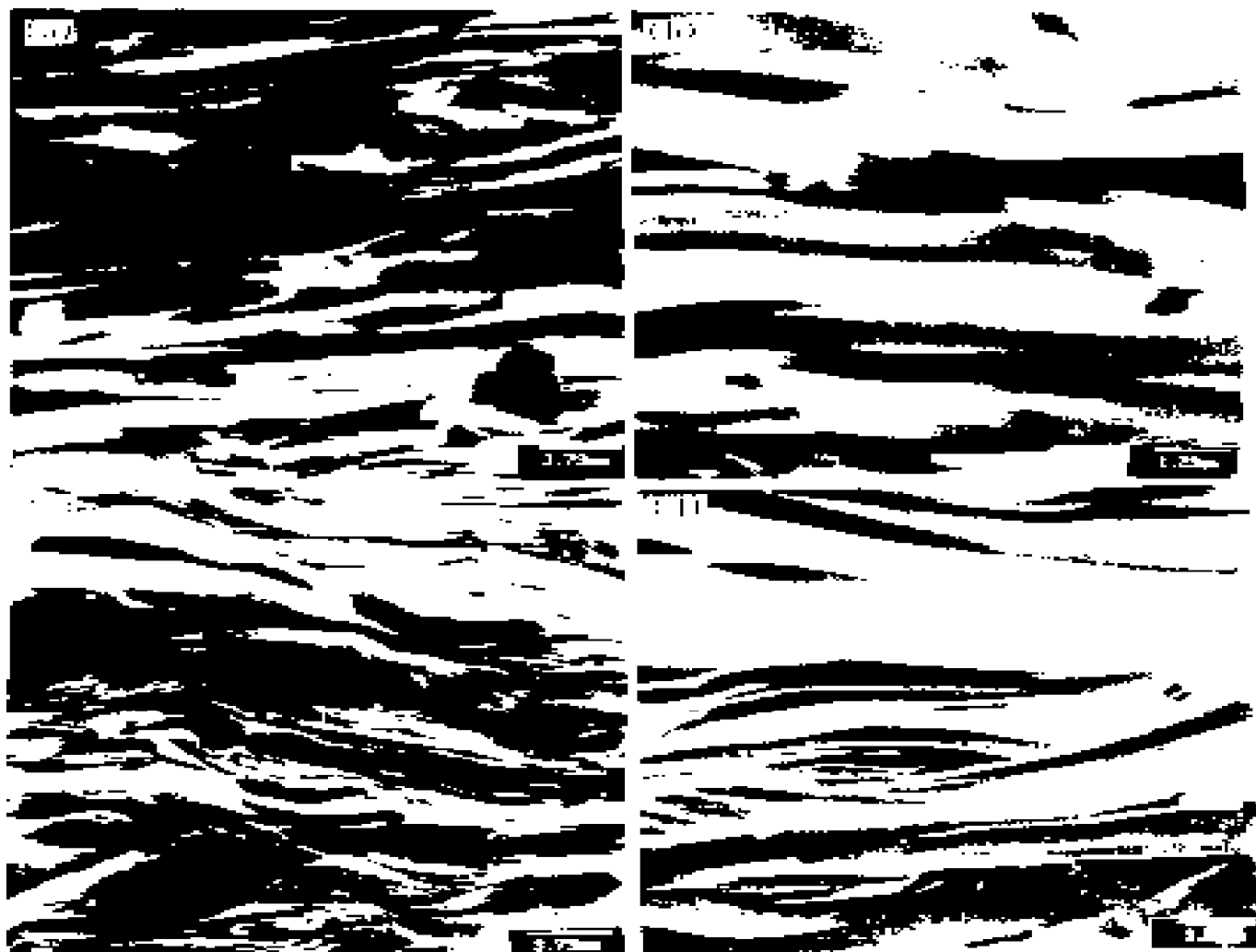
**Fig. 2 HP reduction ratio versus critical current density curves**

The tapes after the process of 838 °C, 50 h+ HP+ 838 °C, 60 h+ HP+ 838 °C, 60 h, HP represents pressure changes with different reduction.

As stated above, the optimum HP parameters are 750 °C, 200 MPa and 30 min, at which the highest  $J_c$  of  $2.25 \times 10^4 \text{ A}/\text{cm}^2$  (77 K, zero field) was obtained.

### 4 ANALYSIS OF HP DEFORMATION MECHANISM

Fig. 3 shows the typical SEM micrographs of longitudinal fracture sections of cold and hot pressed tapes. The zero-field values of the critical current was 10.8 A for both tapes. It can be seen that the grains of the cold and hot pressed tapes are both well aligned. However, for the



**Fig. 3 SEM micrographs from longitudinal cross sections**

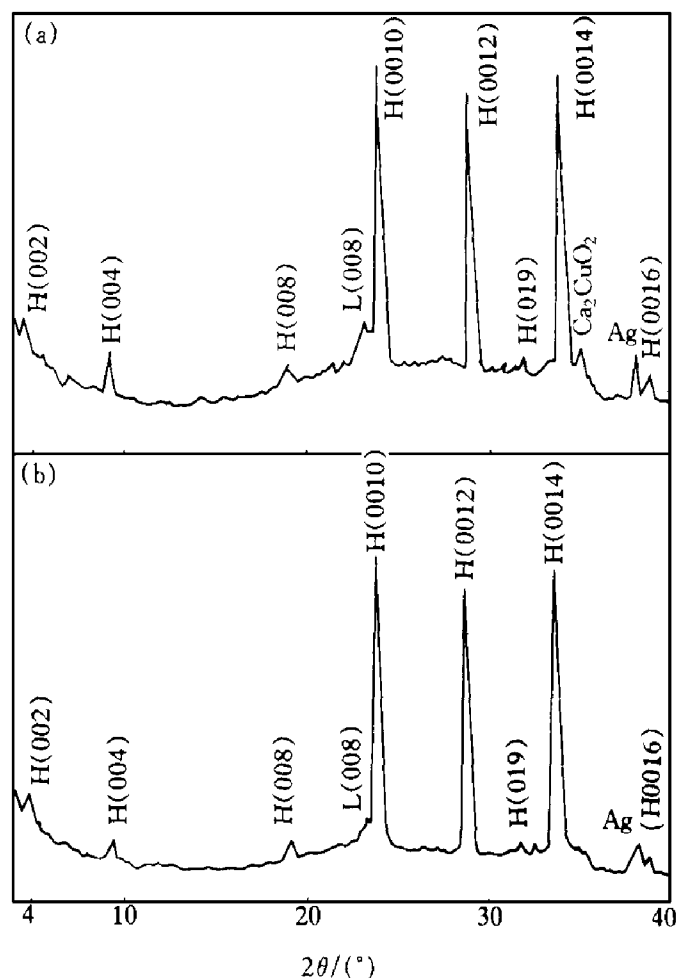
- (a) —Cold pressed tapes after process of 838 °C, 60 h+ CP+ 838 °C, 60 h+ CP+ 838 °C, 60 h (CP: cold pressing); (b) —Magnified view of (a);  
(c) —Hot pressed tapes after the process of 838 °C, 60 h+ HP+ 838 °C, 60 h (HP: hot pressing); (d) —Magnified view of (c)

hot pressed tapes there are characteristic clusters of grains which bond together to form dense microstructure of well-aligned grains (Fig. 3(c)), voids are rarely observed between the crystals, and several secondary phases are present to the tapes, being involved closely by the grains. The difference in the microstructure between the cold and hot pressed tapes is that the hot pressed tapes underwent sufficient densification at high temperatures. In fact, the degree of grain twisting is obviously strong in the hot pressed tapes (Fig. 4(d)), which may be attributed to the softening of grains during hot deformation. According to the previous reports<sup>[8-9]</sup>, the grains with a misorientation angle of over 5~10° great-

ly reduced  $J_c$ . Therefore, the misorientation characteristic of grains resulted from hot pressing is very harmful to the improvement of  $J_c$  at 77 K, zero field.

X-ray diffraction measurements of two specimens are given in Fig. 4 which shows that the intensities of the [001] peaks of the Bi(2223) phase are strong, which indicates the presence of preferentially oriented Bi(2223) grains after heat treatment and pressing cycles. It also confirms that the peaks of secondary phases for HP samples are mainly  $\text{Ca}_2\text{Cu}_3\text{O}$ . The degree of texture ( $F$ ) was defined by the Lotgering method<sup>[10]</sup> as  $F = (P - P_0)/(1 - P_0)$ , where  $P = \sum(00l)/\sum(hkl)$ .  $P$  is the sum of integrated

intensities for all (00l) reflections divided by the sum of all intensities for (hkl) reflections in the oriented specimen,  $P_0$  is an equivalent parameter for a randomly oriented specimens. For HP and CP specimens the degrees of texture are 0.90 and 0.88, respectively. This demonstrates that the effects of HP and CP on the tape core are very similar, both enhance the degree of texture.



**Fig. 4 X-ray diffraction patterns of cold and hot pressed tapes in Fig. 4**

(a) —Hot pressing; (b) —Cold pressing

It is well known that initial densification occurs by sliding and fragmentation of particles. In cold pressed tapes the density of superconductor is increased under suppression. But hot pressing caused densification via pressure induced volume diffusion at higher temperatures. In other words, the increase of liquid formation and the stresses present at the particle contacts, adding plastic yielding, enhance density during hot pressing, because an intergranular liquid phase plays an important role in the diffusion<sup>[11]</sup>. The

experiment confirms that during hot pressing, deformation resistance of the tape decreases significantly, and the large reduction is obtained under low pressure. The pressure of hot pressing is about 10% of that of cold pressing to achieve the same reduction.

## 5 CONCLUSIONS

(1) The optimum HP parameters are 750 °C, 200 MPa and 30 min at 77 K and zero field, the maximum  $J_c$  value is up to  $2.25 \times 10^4$  A/cm<sup>2</sup>.

(2) Hot pressing can increase the density of superconducting core effectively. In comparison with cold pressing, hot pressing can obtain larger reduction ratio under low pressure.

(3) Because of the softening of grains during hot deformation, grains with a misorientation angle often turn up, and this is harmful to the improvement of  $J_c$  at 77 K and zero field.

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